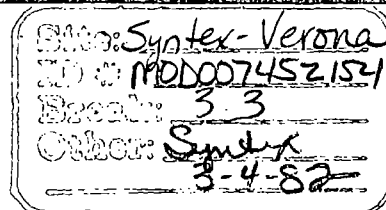




MEMO TO: L. Throop

DATE: A/R 3856
March 4, 1982COPIES TO: A. Holstein, J. Fried
B. Brunck I. Nordlicht
C. Sawyer K. Stormer, B. Spence, J. MurrayFROM: D. DeiRossi *DDR*SUBJECT: METHOD VALIDATION FOR DETERMINATION OF 2,3,7,8-
TETRACHLORODIBENZO-P-DIOXIN (TCDD) IN SOIL

Four sediment samples from Flat Creek adjacent to the Denney Farm Site have been analyzed by the method "Determination of 2,3,7,8-TCDD in Soil" (A/R 3643). Three samples were spiked with ^{14}C -TCDD at a concentration level of approximately 10, 20, and 30 ppt, respectively. A fourth sample was unspiked. The spiking solution of approximately 0.216 ng/ml ^{14}C -TCDD was used also as the external standard for the Capillary-Electron Capture-Gas Chromatography analyses. The results which confirm the capability of the method to detect 2,3,7,8-TCDD at a concentration level of 10 ppt are shown in Table 1. The recovery values determined by scintillation counting show the lowest recovery value to be 92.5%. The maximum relative error of 21.0% is considered acceptable for analysis at this level of detection.

Table 1

<u>Actual Spike Level (ppt)</u>	<u>Recovery of ^{14}C-TCDD</u>	<u>Found Spike Level (ppt)</u>	<u>Relative Error</u>
10.8	93.1%	12.6	16.7%
21.6	97.3%	23.3	7.9%
32.4	92.5%	39.2	21.0%

Two GC injections were made for each sample. The peak heights corrected for recovery are plotted versus the ^{14}C -TCDD spike content for each of the four samples in Graph 1. The y-intercept of 1.92 mm, estimated by a least squares linear regression, is consistent with peak heights of 3.0 mm and 3.25 mm for the blank analysis and for a noise level of approximately 3 mm in the TCDD retention time region shown in Chromatograph 1. The peak heights of 10.5 mm and 11.5 mm for the 10 ppt spiked sample thus give a signal to noise ratio of at least 3.5. Chromatogram 2 shows a representative GC trace for the 10 ppt sample.

Notebook Reference: Dale DeiRossi 6725-91 to 99 and 122-133

j/

40028090
SUPERFUND RECORDS

PEAK HEIGHT (Corrected for Recovery)

40

30

20

10

Graph #1

Peak Height (Corrected for Recovery)

V.S. "4C-2,3,7,8-TCDD" Spike Amount

NUMBER OF KNOWN POINTS

? 8

X,Y OF POINT1 ? 0,3.5

X,Y OF POINT2 ? 0,3.25

X,Y OF POINT3 ? 0.216,11.28

X,Y OF POINT4 ? 0.216,12.35

X,Y OF POINT5 ? 0.432,21.07

X,Y OF POINT6 ? 0.432,22.61

X,Y OF POINT7 ? 0.648,34.59

X,Y OF POINT8 ? 0.648,38.92

$F(X) = 1.9215045 + (51.0023011 * X)$

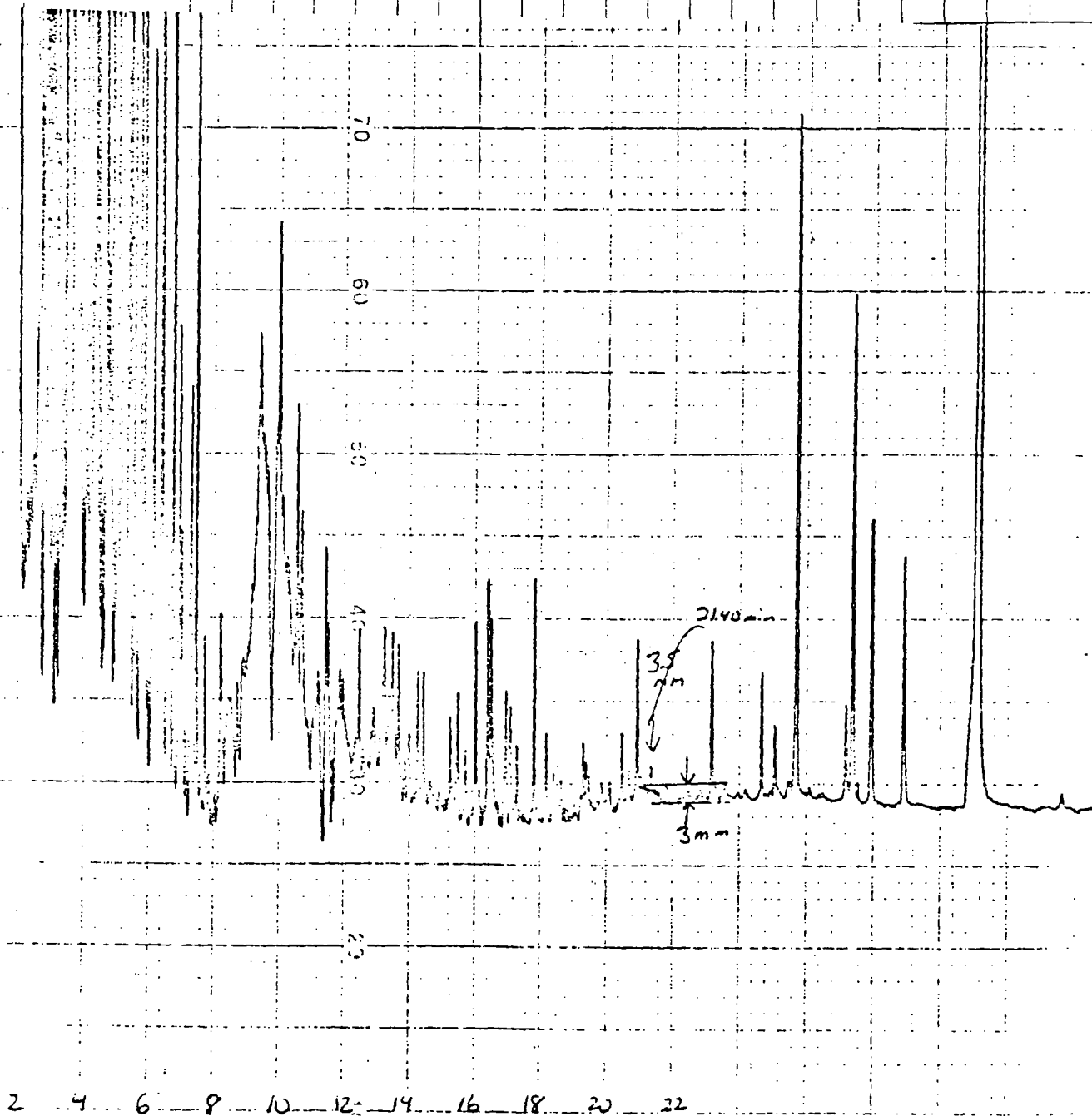
COEFFICIENT OF DETERMINATION (R^2) = 0.973357285

COEFFICIENT OF CORRELATION = 0.9865887

STANDARD ERROR OF ESTIMATE = 2.352995

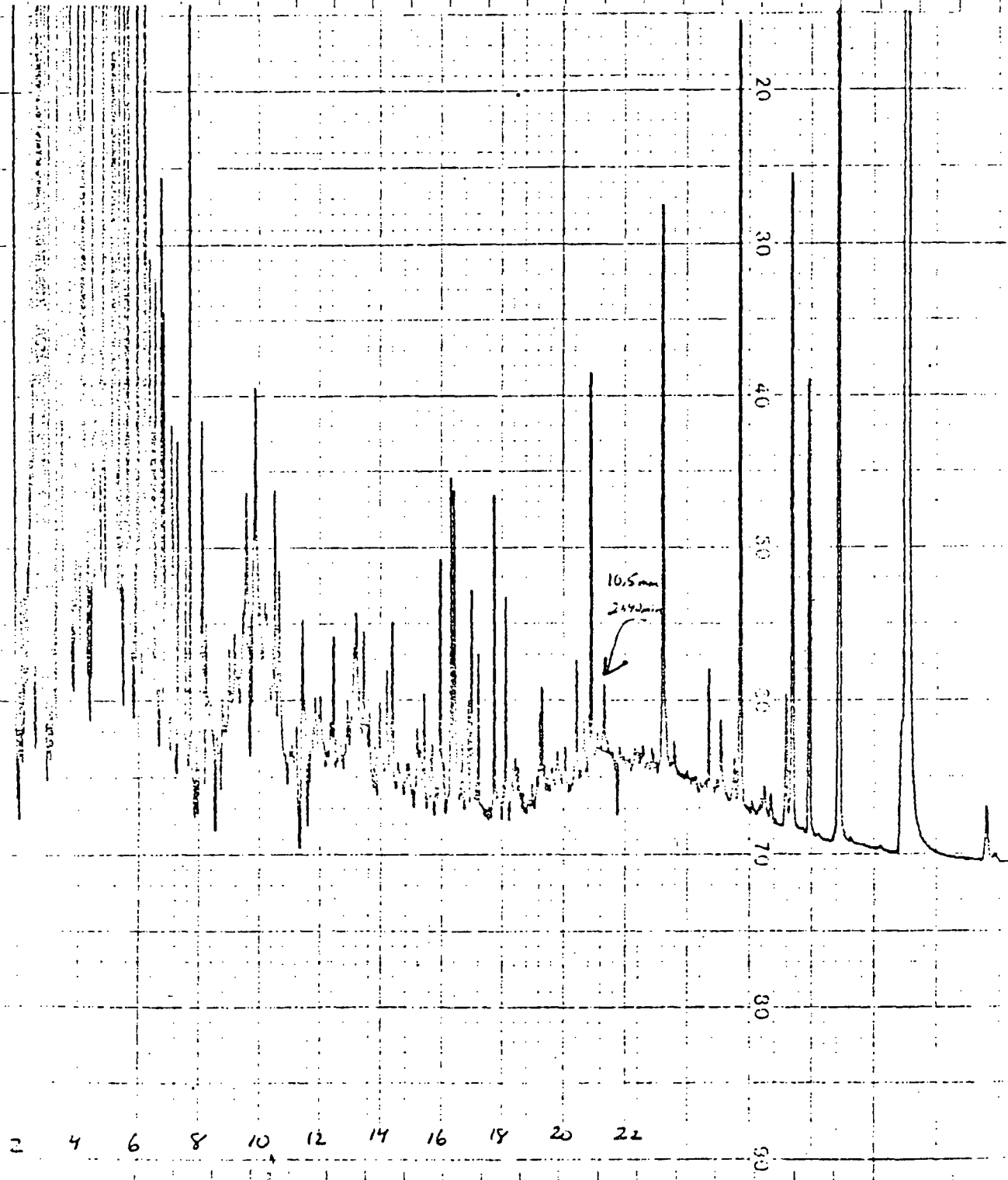
CHROMATOGRAM #1- BLANK

2.028 6725-91-2 in 1ml toluene
X16 2/1/82 ORR



CHROMATOGRAM #2- 10 PPT SPIKE

and 6795-961 added in 1st taken





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DATE 10/8/92

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KCE	KC 100	

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NO. PCS.	DESCRIPTION	WEIGHT	CHARGE
	K-10V		

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- ☐ W.W.G.
☐ B.D.

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